

**What Is Claimed Is:**

1. A method for preparing expandable graphite flake exhibiting one or more improved exfoliation characteristics, which comprises:

- 5 (a) contacting graphite flake with an organic expansion aid;
- (b) subjecting said graphite flake to an electrolytic oxidation treatment with an aqueous intercalant solution to provide intercalated graphite flake; and
- (c) recovering said intercalated graphite flake.

10 2. A method according to claim 1 wherein the graphite flake is contacted with said expansion aid prior to subjecting said graphite flake to electrolytic oxidation.

3. A method according to claim 1 wherein the graphite flake is contacted with said expansion aid by dissolving said expansion aid in said aqueous intercalant solution prior to  
15 subjecting said graphite flake to electrolytic oxidation therein.

4. A method according to claim 1 wherein said aqueous intercalant solution comprises sulfuric acid.

20 5. A method according to claim 1 wherein said expansion aid comprises a carboxylic acid soluble in said aqueous intercalant solution in an amount effective to enhance exfoliation.

6. A method according to claim 5 wherein said carboxylic acid comprises a carboxylic  
25 acid selected from the group consisting of lower aliphatic carboxylic acids and dicarboxylic acids and mixtures of these.

7. A method according to claim 6 wherein said acid comprises a carboxylic acid of the formula  $H(CH_2)_nCOOH$  wherein  $n$  is a number of from 0 to about 5.

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8. A method according to claim 1 wherein said intercalant solution contains from about 30 to about 85% water by weight of the solution.

9. A method according to claim 8 wherein said intercalant solution contains from about 50 to about 75% water by weight of the solution.

10. A method according to claim 1 wherein said aqueous intercalant solution comprises from about 10 to 75% sulfuric acid, said expansion aid comprises an amount effective to enhance exfoliation of from about 1 to 10% of a carboxylic acid soluble in said aqueous intercalant solution, and said intercalant solution contains from about 30 to about 85% water, all percentages based on the weight of the solution.

11. A method according to claim 1 wherein the electrolytic oxidation treatment comprises passing a current between a cathode and the graphite flakes as an anode at an anode current density of from about 0.02 to about 0.06 amps per square centimeter.

12. A method according to claim 1 wherein the electrolytic oxidation treatment comprises passing a current between a cathode and the intercalant wet graphite flakes as an anode at a cell voltage of from about 1 to about 6 volts.

13. A method for preparing expandable graphite flake exhibiting one or more improved exfoliation characteristics, which comprises:

(a) contacting graphite flake with an organic expansion aid comprising a carboxylic acid selected from the group consisting of lower aliphatic carboxylic acids and dicarboxylic acids and mixtures of these, said contacting being prior to subjecting said graphite flake to electrolytic treatment;

(b) then, subjecting said graphite flake to an electrolytic oxidation treatment using an aqueous, intercalant solution to provide intercalated graphite flake, by passing a current through the solution between a cathode and graphite flake wet with the intercalant as

an anode at a current density of from about 0.02 to about 0.06 amps per square centimeter and at a cell voltage of from about 1 to about 6 volts; and

(c) recovering said intercalated graphite flake.

5 14. A method according to claim 13 wherein said aqueous intercalant solution comprises from about 10 to 75% sulfuric acid and said expansion aid comprises a carboxylic acid soluble in said aqueous intercalant solution and is employed in an amount of from about 1 to 10%, both percentages based on the weight of the intercalant solution.

10 15. A method for preparing expandable graphite flake exhibiting one or more improved exfoliation characteristics, which comprises:

(a) subjecting said graphite flake to an electrolytic oxidation treatment by passing a current through the solution between a cathode and the graphite flake as an anode at a current density of from about 0.02 to about 0.06 amps per square centimeter and at a cell voltage of from about 1 to about 6 volts with an aqueous intercalant solution containing  
15 from about 10 to 75% sulfuric acid based on the weight of the solution and an organic expansion aid in an amount of from about 1 to 10%, to provide intercalated graphite flake; and

(b) recovering said intercalated graphite flake.

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16. Intercalated graphite flake prepared according to the process of claim 1.

17. Intercalated graphite flake prepared according to the process of claim 10.

25 18. Intercalated graphite flake prepared according to the process of claim 13.

19. Intercalated graphite flake prepared according to the process of claim 15.

30 20. Intercalated graphite flake prepared according to the process of claim 1, characterized by an intumescent temperature of below about 200°C.

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